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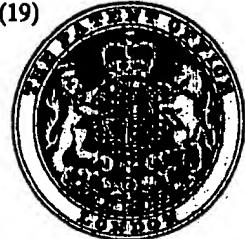
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(19)



(54) GASEOUS MIXTURE FOR USE IN HEAT TREATMENT OF METALS

(71) We, BOC LIMITED, of Hammersmith House, London W6 9DX, England, an English company, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:

This invention relates to a gaseous mixture for use in the heat treatment of metal (typically, ferrous metal). In particular, it relates to a method of forming a gaseous mixture suitable for use in the heat treatment of ferrous or non-ferrous metal and to apparatus for forming such mixtures.

Examples of processes of heat treatment of metals include carburising, carbonitriding, nitro carburising, carbon restoration, case hardening, neutral hardening, decarburising, malleabilising, annealing and normalising.

In order to heat treat metal it is standard practice to provide in the furnace in which the treatment is performed a non-oxidising atmosphere (except when on occasions it is desired to oxidise the metal). The gas used to provide such an atmosphere is often referred to as "carrier gas". For all types of heat treatment listed above, it may be necessary at some stage of the treatment to provide in the furnace an atmosphere of chosen "carbon potential". The term "carbon potential" applied to an atmosphere in a heat treatment furnace indicates the percentage carbon content that the metal in the furnace would have were the metal and atmosphere to come to equilibrium with each other. It is to be appreciated that in heat treatment the atmospheres in the furnace is generally not equilibrium with the metal.

It is frequently the practice to form an atmosphere of controlled carbon potential by adding methane or other hydrocarbon gas or vapour to the carrier gas. When

carbonitriding ammonia in addition to the hydrocarbon is added to the carrier gas.

There are a number of well known ways of forming a suitable carrier gas. The most commonly practiced is to pass fuel gas and air through a so called "endothermic generator". In such a generator a catalyst is provided and by keeping the temperature therein at a suitable value (eg. 1050°C) the fuel gas and air react catalytically to form a gas mixture typically consisting of about 24% by volume of carbon monoxide, about 30% by volume of hydrogen up to 1% of each of carbon-dioxide, methane and water vapour, and a balance of nitrogen. It is necessary to heat the gases in the generator as the reaction does not release sufficient heat to the nitrogen in the air, (requires heat to keep it going).

A second well known method of forming gas suitable for heat treatment of metals involves burning fuel gas in a so-called exothermic generator. This procedure results in the formation of greater proportions of carbon dioxide and water vapour than are produced in an endothermic generator.

Both exothermic and endothermic generators suffer from the disadvantage that they are relatively costly equipment and that they occupy considerable floor space in the foundry or heat treatment shop where they are used. Moreover, there are considerable operating costs associated with the cooling or heating of the generators.

A third well known way of forming a furnace atmosphere is to react together in the furnace regulated proportions of hydrocarbon and oxygen-containing gas or vapour. This is generally done by mixing the hydrocarbon and oxygen-containing gas or vapour together with nitrogen outside the furnace and passing a mixture into the furnace. Once inside the heated furnace the mixture reacts to form the carbon monoxide that is necessary for carburising to take

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- place. This method has the advantage of avoiding exothermic and endothermic generators. However, the gas supply, and mixing equipment requires careful calibration as a change in, for example, relative proportions of fuel gas in the mixture supplied to the furnace will effect the carbon potential of the atmosphere inside the furnace in a complex way. There is also a limit to the level of CO which can be formed by this method which may lead to uneven case depths.
- According to the present invention there is provided a method of forming a gaseous mixture suitable for use in the heat treatment of metals, which method comprises reacting oxygen (or oxygen-enriched air containing at least 50% by volume of oxygen) or preheated air and a combustible hydrocarbon in the reaction zone of at least one reactor to produce gaseous products comprising hydrogen and carbon monoxide, the reaction zone of the or each reactor being situated or terminating in a passage or chamber which is in communication with the gas space in the furnace in which the heat treatment is performed and which is isolated from the ambient air, or being situated, or terminating in the furnace chamber itself, and mixing inert gas (as herein defined) with the gaseous products.
- By the term 'inert gas' as used herein is meant nitrogen or a noble gas such as argon krypton or helium.
- By this means only the preheated air, oxygen or oxygen-enriched air supplied to the or each reactor is used to support the reaction.
- The invention also provides apparatus for forming a gaseous mixture suitable for use in the heat treatment of metals, which apparatus comprises at least one reactor capable of reacting oxygen (or oxygen-enriched air containing at least 50% by volume of oxygen) or preheated air with a combustible hydrocarbon in its reaction zone to produce gaseous products comprising hydrogen and carbon monoxide the or each reactor being situated, or terminating, in a passage which is in communication with the gas space in the furnace in which the heat treatment is performed and which is isolated from the ambient air, or being situated, or terminating, in the furnace itself, and means for mixing the gaseous products with inert gas (as herein defined).
- In general, because of the chemical equilibria involved, it will be impossible to avoid formation of some carbon dioxide and water vapour. Typically up to 3% by volume of carbon dioxide may be formed.
- Preferably, oxygen rather than oxygen-enriched air is supplied to the or each reactor. This makes sure that there is no nitrogen supplied with the oxygen to the reaction zone of the or each reactor. Such nitrogen acts as a coolant. Indeed, in conventional endothermic gas generators it is the presence of nitrogen in admixture with the air which results in the large heat input requirements of endothermic gas generators. We believe that the reaction between oxygen and a fuel gas such as methane is self-sustaining without the input of this heat. However, if desired the oxygen, oxygen-enriched air or preheated air may be preheated to the temperature of the furnace.
- Moreover, even though with oxygen-enriched air there would be nitrogen which has a cooling effect, this cooling effect will not be marked as with air and we believe that the oxygen-enriched air containing at least 50% by volume of oxygen it would be possible to operate each reactor effectively without the input of heat or with the input of only a relatively small amount of heat (ie. that provided by a small electro-resistive coil in the reaction region or zone). Nonetheless, if oxygen-enriched air is chosen it preferably contains at least 80% of oxygen and more preferably at least 90% of oxygen (these percentages being by volume).
- Alternatively, air, unenriched in oxygen, may be used instead of oxygen or oxygen-enriched air, if the air is preheated. It is possible to use heating means provided in the furnace for raising the temperature therein to perform also the duty of preheating the air, thus avoiding the need to have an additional source of heat.
- The hydrocarbon is preferably selected from methane, ethane, propane and butane. Methane is particularly preferred. So is propane.
- The relative rates of supply of the hydrocarbon and the oxygen, oxygen-enriched air or air to the or each reactor (or burner) are preferably such that the hydrocarbon is oxidised to carbon monoxide and hydrogen with formation of only traces of carbon dioxide and water vapour. This means that the oxygen, oxygen-enriched air or air and hydrocarbon should be preferably supplied in the stoichiometric quantities necessary for this to happen. This, if the hydrocarbon is methane, 2 volumes of methane per unit time should be supplied to the combustion region of the or each burner for each volume per unit time of oxygen.
- If desired, irrespective of what the hydrocarbon is, a small excess of oxygen or oxygen-enriched air may be supplied to the reaction of one of the or each reactors so as to make possible the formation of carbon dioxide (and water vapour if the carbon containing substance is a hydrocarbon) in quantities larger than 3% by volume.
- However, if for any reason, the presence of either water vapour or carbon dioxide is

- required in the furnace, metered quantities of these substances may be injected into the furnace or into the passage downstream of the or each burner.
- Preferably, the inert gas (for example nitrogen) is supplied to the furnace via the passage. Instead of nitrogen, an inert gas such as argon, krypton or helium may be supplied to the furnace via the passage. This makes possible the supply to the furnace of a gas mixture which contains a greater proportion of nitrogen than gas from endothermic generators. This has the advantage of reducing the flammability of the gas mixtures supplied to the furnace.
- The or each reactor may be situated in the furnace itself, preheating air used to react with the hydrocarbon and inert gas supplied separately to the furnace, and the gases in the furnace mixed by means of, for example, a fan.
- Methane or other fuel gas may be supplied to the furnace directly or via the passage when it is desired to perform a carburisation or like heat treatment process. In addition, when carbonitriding ammonia may in addition to methane be supplied to the furnace either directly or via the passage. If methane and/or ammonia are supplied via the chamber or passage, each is preferably injected into the passage downstream of the reaction zone of the or each burner. Preferably, each gas to be introduced into the reactor and passage or chamber furnace is supplied through a flow meter or calibrated orifice at a chosen flow rate. It is possible to vary the composition of the gas mixture in the furnace quite widely. For example, the relative proportions of nitrogen (or other inert gas), hydrogen and carbon monoxide may be varied widely by varying the rate at which the nitrogen is supplied to the passage. Preferably, the gas mixture supplied to the furnace contains at least 70% by volume of nitrogen. It is not, however, desirable to vary the relative proportions of oxygen, oxygen-enriched air or preheated air and hydrocarbon supplied to the or each reactor. However, some flexibility in the relative proportions of the reaction products of the or each reactor may be introduced without producing carbon dioxide in these reaction products by changing the hydrocarbon supplied to the or each reactor and adjusting the rates of supply of the oxygen or oxygen-enriched air and the hydrocarbon accordingly. For example, the ratio of carbon monoxide to hydrogen may be increased from 1:2 to 3:4 (proportions by volume) by substituting propane for methane and increasing threefold the ratio of the rates at which molecular oxygen and hydrocarbon are passed to the reaction zone or the r each reactor.
- Alternatively, ratios of carbon monoxide to hydrogen between the two quoted may be achieved by using two or more reactors, at least one of which is supplied with methane. In general, the relative proportions in which the gases should be supplied should be chosen in accordance with the heat treatment process being performed.
- It is alternatively possible to synthesise a gas mixture similar in composition to the gas produced by conventional operation of an endothermic generator.
- The criteria for choosing the appropriate mixture are well known in the art. The present invention offers a system of providing the appropriate mixture which in its operation is analogous to that of an endothermic gas generator so that little difficulty should be presented in the control of the carbon potential of the atmosphere in the furnace.
- The gases supplied to the or each reactor are preferably not pre-mixed before being introduced in to the reaction zone.
- The or each reactor may be similar in construction to a conventional oxy-fuel gas burner. Indeed, such a burner may be used as the or each reactor. Few (if any) modifications will be required to the burner in order to use it in the method and apparatus according to the present invention.
- However, it is preferred that its nozzle or end be fitted with or have a small vessel, (having an outlet) in which the reaction zone is isolated from the gas space in the passage surrounding it so as to prevent any gas from this space becoming entrained in the zone. This will reduce the quenching effect that gas passing through the passage may have on the reaction. The vessel may contain a catalyst such as nickel. Alternatively the vessel or nozzle of the reactor may itself be of nickel. However, neither of these expedients is essential. It is also desirable for the reactor to be fitted with a conventional flashback arrestor. Other preferred features for the or each reactor are an ignition device for initiating the reaction between the carbon-containing substance and the oxygen or oxygen-enriched air.
- For example, a piezoelectric device may be used for this purpose as in a conventional burner. In addition, it is desirable for the burner to have some sort of device or means provided to make possible detection of whether or not the reaction between the oxygen or oxygen-enriched air and the hydrocarbon or other carbon containing material is taking place. Thus, some property of the combustion may be monitored; for example, a thermo couple may detect the absence or presence of the reaction by virtue of the different temperatures it records according to whether or not there is a reaction taking place. Alternatively, a photoelectric cell or like device may detect

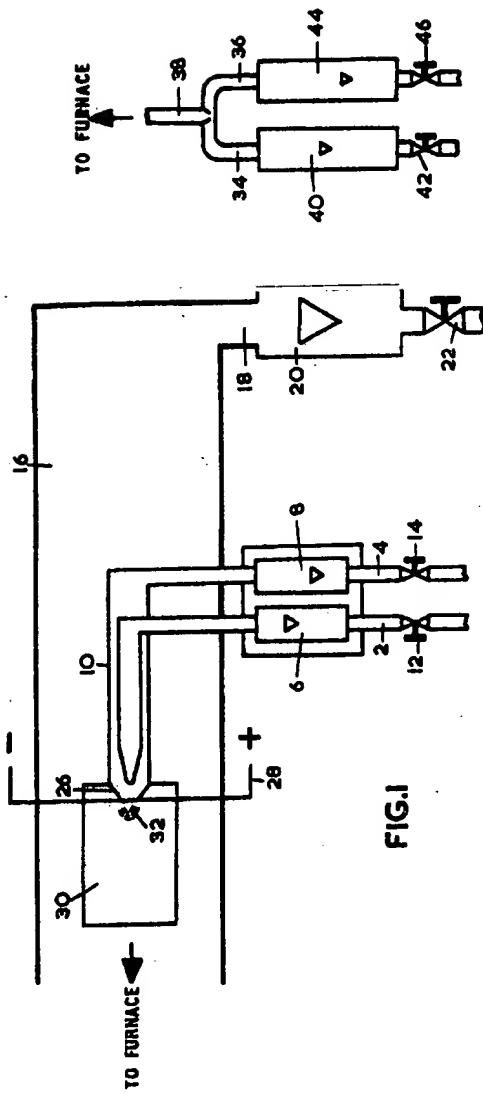
- the difference in the quantity in the light falling upon it when there is or is not a reaction taking place.
- Another alternative is a gas detector which is capable of detecting the presence or absence of, say, carbon monoxide or hydrogen in the intended reaction zone. Alternatively, the passage may be provided with a window whereby an observer may view the reaction of the or each reactor and thereby be able to detect visually whether or not there is a reaction going on. Preferably, supply of the oxygen, oxygen-enriched air or preheated air and hydrocarbon or other carbon-containing substances is arranged such that if the absence of a reaction there between is detected by the detector a signal is generated such that valves (eg. solenoid-controlled valves) which control the passage of the gases to the reactor (or each reactor) are closed automatically. This avoids any damage to the furnace or risk of explosion that may be caused were oxygen to be introduced into the furnace atmospheres.
- It is desirable that the reactor be of a type which does not cause appreciable volumes of gas to be entrained from the surrounding atmosphere into the reaction region. Generally such a reactor will have a relatively long nozzle and may be in general similar in construction to a conventional oxy-fuel burner. We prefer to call such a device a reactor as the oxygen heat of the carbon monoxide forming reaction between 1 mole of methane and 1/2 mole of oxygen is approximately 8 kcal. This (incomplete combustion) does not constitute a large evolution of heat normally associated with combustion.
- It is desirable for the or each reactor to be disposed such that its axis is parallel to or coaxial with the axis of the passage.
- Instead of or in addition to having a detector for detecting whether or not the reaction between the oxygen and the hydrocarbon is taking place, the or each burner may have a small electrical heater associated with the outlet of its nozzle, which heater may be operated continuously. Should for any reason the flame fail the heater will be effective to restart the reaction.
- The invention will be described by way of example with reference to the accompanying drawings of which.
- Figure 1* is a diagram schematic representing a gas supply apparatus for supplying a gas mixture to a heat treatment furnace, and
- Figure 2* is a diagram schematically representing a furnace fitted with apparatus for forming therein a gas mixture suitable for use in the heat treatment of metals.
- Referring to Figure 1 of the drawings, equipment for controlling the supply of gas to a heat treatment furnace (not shown) is illustrated. The apparatus includes an oxygen supply line 2 and a hydrocarbon supply line 4. In the supply lines 2 and 4 are flow meters 6 and 8 respectively. The supply line 2 is in communication with an inner annular passage of a reactor 10 similar in construction to a conventional oxy-fuel burner. The supply line 4 is in communication with an outer annular passage of the burner 10. Also in the supply line 2 upstream of the flow meter 6 is a solenoid-operated manual flow-control valve 12. A similar valve 14 is located in the supply line 4 of the flow meter 8.
- The reactor 10 is coaxial with a passage 16. One end of the passage 16 terminates in the gas space of the furnace (not shown) the other end of the passage communicates with a nitrogen supply line 18. In the nitrogen supply line 18 is disposed a flow meter 20, and upstream of the flow meter 20, a solenoid-operated manual flow control valve 22.
- The reactor 10 has a nozzle 26 with whose outlet is associated a piezo electric ignition device 28 and a small chamber 30 (having a gas outlet) which may contain a nickel catalyst. There is also a reaction sensor or detector 32 located in what it intended to be the reaction (or combustion) zone of the reactor 10.
- The illustrated equipment also includes a hydrocarbon supply line 34 and an ammonia supply line 36 connected to a common inlet 38 which terminates directly in the gas space of the furnace (not shown). In the conduit 34 is a flow meter 40 and upstream of the flow meter 40, a solenoid-operated or manual flow control valve 42. In the ammonia supply line 36 is a flow meter 44 and, upstream of the flow meter 44, a solenoid-operated flow control valve 46.
- In order to supply a gas mixture to the furnace, line 2 is connected to a source of oxygen (not shown) and the line 4 is connected to a source of hydrocarbon (for example methane). In addition, the line 18 is connected to a source of nitrogen. These sources are not shown in the drawings. These sources may be cylinders containing the respective gases under pressure in the liquefied state. If the latter is the case, the cylinder (or other contained) may have associated therewith a vaporiser for vaporising the liquefied gas. If it is desired to supply oxygen-enriched air instead of oxygen this gas may be supplied from a plane operating a "pressure swing" adsorption-desorption cycle.
- The flow meters 6, 8 and 20 may be set in accordance with the desired composition for the carrier gas mixture. If, for example, it is required to supply a carrier gas mixture at a rate of 300 standard cubic feet per hour of a carrier gas mixture comprising 80% by volume of nitrogen, 13.33% by volume of hydrogen and 6.66% by volume of carbon

- monoxide, the flow meter 20 may be set so as to pass nitrogen therethrough at a rate of 20 standard cubic feet per hour, and the flow meter 6 set so as to pass oxygen therethrough at a rate of 10 standard cubic feet per hour.
- If it is desired to perform a carburising, the supply line 34 may be connected to a source of hydro-carbon, for example, methane, and the flow meter 40 set to pass methane into the furnace at a chosen rate. The supply of methane to the furnace via the common inlet 38 may then start.
- When it is desired to perform a carbonitriding a source of ammonia may be connected to the line 36, the flow meter 44 given the appropriate setting and a supply of ammonia started in addition to that of the methane through the line 34.
- In the reaction zone of the reactor the oxygen and methane supplied thereto react to form hydrogen and carbon monoxide. A gas mixture consisting of nitrogen, hydrogen and carbon monoxide therefore enters the furnace, via the passage 16. To start the reaction the ignition device 28 may be operated. Should the reaction fail, this may be detected by the sensor 32 which, if desired, may be connected in circuit with the solenoid valves 12, 14, 22, 42 and 46 such that if the reaction does fail all the solid valves are closed, thereby shutting off the supply of gas to the furnace.
- Referring to Figure 2 of the drawings, a heat treatment furnace 60 is shown. Situated in the furnace 60 is a reactor 62. The reactor 62 is substantially the same as the reactor 10. It is however in communication with a source (not shown) of air rather than a source of oxygen.
- The reactor 62 is able to be supplied with hydrocarbon via a pipeline 68 having disposed in it a flow meter 64 and upstream of the flowmeter 64 a solenoid or manually-operable flow control valve 66.
- The reactor 62 is able to be supplied with air via a pipeline 74 having disposed in it a flow meter 70 and upstream of the flowmeter 70 a solenoid or manually-operable flow control valve 72. The pipeline 74 downstream of the flow meter 70 is in heat exchange relationship with a heating tube or element 76 inside the furnace 60.
- The furnace 60 has a gas supply pipeline 78 terminating in communication with its interior. The pipeline 78 is able to be fed with gas by two subsidiary pipelines 80 and 82. The pipeline 80 is in communication with a source (not shown) of nitrogen. The pipeline 82 is in communication with a source (not shown) of hydrocarbon. In the pipeline 80 is a flowmeter 84 and upstream of the flowmeter 84 a solenoid or manually-operable valve 86. In the pipeline 82 is a flowmeter 90 and upstream of the flowmeter 90 a solenoid or manually-operable valve 92.
- In order to mix the gases supplied to the furnace 60 a fan 88 is provided therein. The apparatus may be operated to form in the furnace a gas mixture or atmosphere of chosen composition.
- With the valves 66 and 72 open, air and hydrocarbon are supplied to the reactor, the air being preheated to 925°C in the furnace, and a reduction between the gases initiated. The valves 66 and 72 are set so as to provide hydrocarbon and oxygen in the stoichiometric proportions necessary for a reaction therebetween to take place yielding carbon monoxide and hydrogen as the reaction products. The extent of the reaction between the oxygen and the hydrocarbon depends on the temperature of the reactants, and can readily be calculated. If desired, it can be arranged for the hydrocarbon supplied to the reactor 62 to be preheated in addition to the air.
- With the valve 86 open nitrogen is supplied to the interior of the furnace via the pipelines 80 and 78. Operation of the fan 88 causes the nitrogen so supplied to mix with the gaseous products issuing from the reactor 62.
- The carbon potential of the furnace atmosphere so created can be adjusted by opening the valve 86 and adding varying quantities of hydrocarbon to the gas mixture in the furnace via the pipelines 72 and 78. If desired, automatic control of the carbon potential may be effected by monitoring the carbon potential in the furnace or a parameter dependent upon the carbon potential by means which generate a signal proportional to the magnitude of the carbon potential or parameter dependent thereupon, said signal generating means being arranged to change approximately the setting of the valve 86.
- WHAT WE CLAIM IS:**
1. A method of forming a gaseous mixture suitable for use in the heat treatment of metals, which method comprises (i) reacting oxygen (or oxygen-enriched air containing at least 50% by volume of oxygen) or preheated air and a combustible hydrocarbon in the reaction zone of at least one reactor to produce gaseous products comprising hydrocarbon and carbon monoxide, the reaction zone of the or each reactor being situated, or terminating, in a passage or chamber which is in communication with the gas space in the furnace in which the heat treatment is performed and which is isolated from the ambient air, or being situated, or terminating, in the furnace chamber itself, and (ii) mixing inert gas (as hereinbefore defined) with the gaseous products.
 2. A method as claimed in claim 1, in

- which the hydrocarbon is selected from methane, ethane, propane and butane.
3. A method as claimed in claim 1 or claim 2, in which the hydrocarbon and the oxygen, oxygen-enriched air or preheated air are supplied at relative rates such that the hydrocarbon is oxidised to carbon monoxide and hydrogen with formation of only traces of carbon dioxide and water vapour.
4. A method as claimed in any one of the preceding claims, in which the inert gas is supplied to the furnace via the passage or chamber.
5. A method as claimed in any one of claims 1 to 3, in which the inert gas is supplied separately to the furnace.
6. A method as claimed in claim 5, in which the oxygen, oxygen-enriched air or air is preheated to the temperature of the furnace.
7. A method as claimed in any one of the preceding claims, in which hydrocarbon is supplied to the furnace separately from the said gaseous products and mixes with the other gases in the furnace.
8. A method as claimed in claim 7, in which the separately supplied hydrocarbon is preheated before it mixes with the other gases in the furnace.
9. A method as claimed in any one of the preceding claims, in which ammonia is supplied to the furnace separately from the said gaseous products, and mixes with the other gases in the furnace.
10. A method as claimed in any one of the preceding claims, in which the gas mixture contains at least 70% by volume of nitrogen.
11. A method as claimed in any one of the preceding claims, in which the or each reactor is an air-fuel gas, or oxy-fuel gas burner.
12. A method as claimed in claim 11, in which the burner has fitted to its outlet end a chamber effective to isolate the reaction zone from the surrounding gas space.
13. A method as claimed in claim 12, in which the chamber fitted to the outlet end of the burner contains a catalyst.
14. A method as claimed in claim 13, in which the catalyst is nickel.
15. A method as claimed in claim 14, in which the burner has a nozzle of nickel.
16. A method of forming a gaseous mixture suitable for use in the heat treatment of metals, substantially as herein described with reference to Figure 1 of the accompanying drawings.
17. A method of forming a gaseous mixture suitable for use in the heat treatment of metals, substantially as herein described with reference to Figure 2 of the accompanying drawings.
18. Apparatus for forming a gaseous mixture suitable for use in the heat treat-
- ment of metals, which apparatus comprises at least one reactor capable of reacting oxygen (or oxygen-enriched air containing at least 50% by volume of oxygen) or preheated air with a combustible hydrocarbon in its reaction zone to produce gaseous products comprising hydrogen and carbon monoxide, the or each reactor being situated, or terminating, in a passage or chamber which is in communication with the gas space in the furnace in which the heat treatment is performed and which is isolated from the ambient air, or being situated, or terminating, in the furnace itself, and means for mixing the gaseous products with inert gas (as hereinbefore defined).
19. Apparatus as claimed in claim 18, in which the passage or chamber is in communication with a source of inert gas.
20. Apparatus as claimed in claim 18, in which a source of inert gas communicates with the furnace separately from the or each reactor, there being a fan in the furnace operable to effect mixing of the inert gas with the gaseous products.
21. Apparatus as claimed in claim 20, additionally including means for preheating the oxygen, oxygen-enriched air or air.
22. Apparatus as claimed in claim 21, in which the preheating means and the or each reactor are situated in the furnace.
23. Apparatus as claimed in any one of claims 18 to 22, in which the or each reactor is an oxy-fuel gas or air-fuel gas burner.
24. Apparatus as claimed in claim 23, in which the burner has fitted to its outlet end a chamber effective to isolate the reaction zone from the surrounding gas space.
25. Apparatus as claimed in claim 24, in which the chamber fitted to the outlet end of the burner contains a catalyst.
26. Apparatus as claimed in claim 25, in which the catalyst is nickel.
27. Apparatus as claimed in claim 23, in which the burner has a nozzle of nickel.
28. Apparatus for forming a gaseous mixture suitable for use in the heat treatment of metals, substantially as herein described with reference to, and as shown in, Figure 1 of the accompanying drawings.
29. Apparatus for forming a gaseous mixture suitable for use in the heat treatment of metals, substantially as herein described with reference to, and as shown in, Figure 2 of the accompanying drawings.

For the Applicants,
M. WICKHAM.

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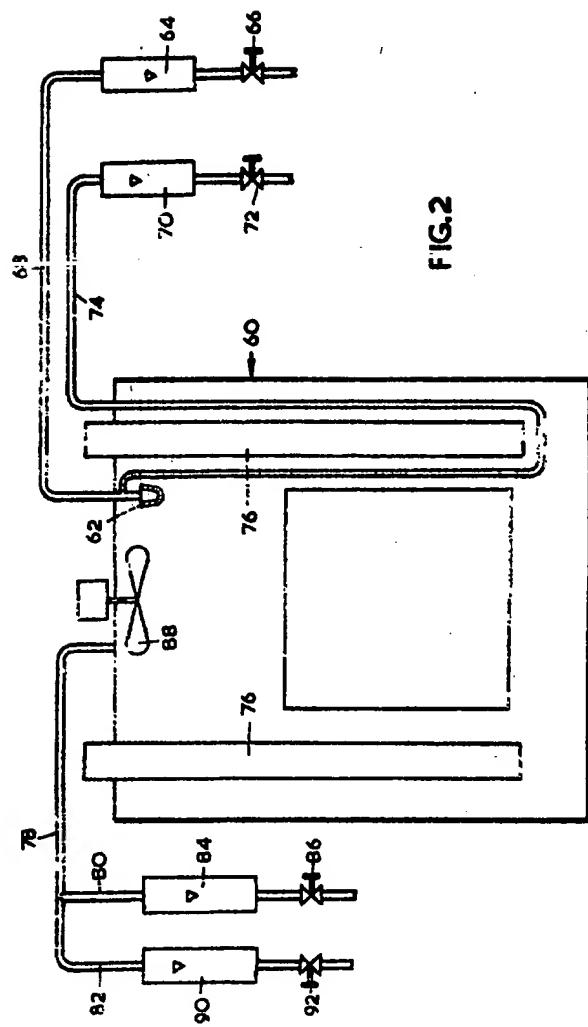


FIG.2